ANALYST:		VPDES NO	

Parameter: Metals
Instrument: _____ Checklist Date_____
06/05

METHOD OF ANALYSIS

	EPA 200 Series (See individual element method no. in 40 CFR Part 136)
	EPA 200.7 Revision 4.4, 1994
	EPA 200.9 Revision 2.2, 1994
	18th Edition of Standard Methods 3000 Series

	SAMPLE COLLECTION AND PRESERVATION	**Y**	**N**
1)	Are all glassware and plasticware acid washed? (Detergent and tap water wash with following rinses: 1:1 HNO_3, tap water, 1:1 HCl, tap water, and final DI water rinse.) If not, were blanks run to ensure acceptability of procedure and materials? [200-4.1; 200.7/200.9-6.10; SM 3010 C.1]		
2)	Are samples for total and total recoverable metals unfiltered and preserved with HNO_3 to a pH of <2? If samples are not preserved in the field, after acidification in the lab are they held for 16 hours before beginning digestion? [40 CFR & 200.9-8.3]		
3)	Are samples for dissolved metals filtered through a 0.45 micron membrane filter immediately after collection and preserved immediately after filtration with HNO_3 to a pH of <2? [40 CFR]		
4)	For dissolved metals, were the filtration apparatus and filter rinsed with 50-100 mLs of sample and the filtrate discarded prior to filtering the aliquot to be preserved as the dissolved metals sample? [200-4.1.1; Permit]		
	SAMPLE PREP		
5)	Are all glassware and plasticware acid washed with detergent and tap water, rinsed with 1:1 HNO_3, tap water, 1:1 HCl, tap water, and final DI water rinse? If not, were blanks run to ensure acceptability of procedure and materials? [200-4.1; 200.7/200.9-6.10; SM 3010 C.1]		
6)	Is the area where metal samples are prepared sufficiently clean to produce data that meet the data quality objectives?]200-5.2.9; SM 3030 A; Permit]		
7)	Are only trace element or equivalent grade reagents used for AA and ICP analysis? [200-7.2; 200.7/200.9-7.1]		
8)	Are reagents being dated and initialed on the container upon receipt and when opened? [Permit]		
9)	DISSOLVED METALS For dissolved metals being analyzed using without digestion, were all of the following true for All Methods? [40 CFR July 1, 1999, footnote 4] COD was low (<20 mg/L) Visibly transparent with turbidity of ≤ 1 NTU Colorless with no perceptible odor Consisted of one liquid phase free of particulate or suspended matter after acidification		
10)	Are digestion fumes and AA exhaust removed by a fume hood or evacuation equipment? [200.7/200.9-5.1; SM3111.6.f]		
11)	Is a digestion log being maintained with the following information recorded? [Permit]		
	a) Date, time, method, and analyst performing digestion.		
	b) Sample ID number with beginning and final volumes.		
	c) pH of sample taken just prior to digestion. NOTE: If pH is > 2, add HNO_3 and hold sample for 16 hrs. Repeat step until verified to be pH < 2. [200.7-8.1; 200.9-8.1]]		
	d) Reagents and volume of each one used.		

	Y	N
e) ID of digestion container used.		
f) Samples used for quality control properly identified [blanks, laboratory control samples (LCS), spikes, and duplicates]		
g) Conc., volume, and source of spiking solution used.		
DIGESTION METHODS		
12) "Less Vigorous Digestion" for Total Metals by FLAA and Sb using GFAA (Preferred method: FR July 1, 1997, footnote 4) Smaller initial sample volumes may be used with acid volumes being adjusted accordingly. [200-4.1.3]		
a) Began with 100 mL of sample?		
b) Added 5 mL of 1:1 HCl?		
c) Volume reduced to 15-20 mL without boiling or going to dryness?		
d) Insoluble materials removed by settling, filtration or centrifugation?		
e) Diluted volume with DI water?		
13) " Less Vigorous Digestion" for Total Metals by GFAA Excluding As, Se, and Sb (Preferred method: FR July 1, 1997, footnote 4) [200 -4.1.4]		
a) As above with HCl OMITTED?		
14) "Vigorous Digestion" for Total Metals by FLAA--NOT for Sb and Sn; insoluble oxides result! Procedure recommended only for samples with very high conc. of organic materials or for colorimetric procedures: FR July 1, 1997, footnote 4. Smaller initial sample volumes may be used with acid volumes being adjusted accordingly. [200-4.1.3]		
a) Began with 100 mL of sample?		
b) Added 3 mL conc HNO_3?		
c) Evaporated to near dryness without boiling or baking sample?		
d) Cooled and added 3 mL conc HNO_3 ?		
e) Covered and refluxed, adding acid until digestion was complete (no additional color change)?		
f) Added 5 mL 1:1 HCl ?		
g) Warmed to dissolve precipitates or residues?		
h) Insoluble materials removed by settling, filtration or centrifugation?		
I) Diluted to volume with DI water?		
15) "Vigorous Digestion" for Total Metals by GFAA--excluding Sn, Sb, As, & Se (FR July 1, 1997, recommends this procedure only for samples with very high amounts of organic materials or for colorimetric procedures.) [200-4.1.3]		
As Above with HCl omitted: substituted HNO_3 for 0.5% final volume?		
16) Digestion for As and Se by GFAA. Smaller initial sample volumes may be used with acid volumes being adjusted accordingly (As -- 206.2; Se -- 270.2)		
a) Began with 100 mL of sample?		
b) Added 1 mL HNO_3?		
c) Added 2 mL 30% H_2O_2?		
d) Reduced volume to slightly less than 50 mL without boiling?		
e) Insoluble materials removed by settling, filtration or centrifugation?		

		Y	N
	f) Diluted to volume with DI water?		
	g) Is appropriate matrix modifier being used?		
17)	**Digestion for methods 200.7 and 200.9.** Smaller initial sample volumes may be used with acid volumes being adjusted accordingly: [11.2]		
	Samples having <1% undissolved solids. a) Began with 100 mL acidified sample?		
	b) Added 2 mL (1+1) HNO_3 and 1.0 mL (1+1) HCl ?		
	c) Volume reduced to 15-20 mL on hot plate without boiling and temperature no higher than 85°C?		
	d) Covered beaker with watch glass and gently refluxed sample for 30 mins.?		
	e) Transferred sample to 50 mL volumetric flask and brought to volume?		
	f) Allowed sample to settle prior to analysis?		
	Samples having >1% undissolved solids: a) Evaporated an aliquot of acidified sample containing no more than 1 g particulate material to approximately 10 mLs.?		
	b) Added 4 mL of (1+1) HNO_3 and 10 mL of (1+4) HCl ?		
	c) Covered with watch glass and gently heated and refluxed for 30 mins.?		
	d) Transferred extract to 100 mL volumetric flask and diluted to volume?		
18)	**Digestion for SM 3030E** (Smaller initial sample volumes may be used with acid volumes being adjusted accordingly): a) Began with 100 mL acidified sample?		
	b) Added 5 mL of conc. HNO_3 and a few boiling beads?		
	c) Brought to slow boil and reduced volume to 10-20 mL?		
	d) Continued heating and adding conc. HNO_3 until digestion was complete?		
	e) Filtered if necessary and transferred to 100 mL volumetric flask and diluted to volume?		
	ANALYTICAL EQUIPMENT		
19)	Is the area where metal samples are prepared sufficiently clean to produce data that meet the data quality objectives? [200-5.2.9; SM3113 B.4.a]		
20)	If micro pipettes are used, are the critical volumes checked quarterly for accuracy and precision? [Permit]		
21)	Are micro pipette tips metal free? (Yellow may have Cd and Cr; Blue may have Cu) [200-5.2.9/6.6]		
22)	Are instrument responses electronically recorded either by strip chart or computer? [200-6.1, 6.5; SM3113 B.4.b]		
23)	Is instrument allowed an appropriate warm-up time? Suggested times are HCL=5-10 min, EDL=30-60 min, and ICP 30-60 min. [200-9.1;200.7/200.9-11.4.3; SM3111 B.4.b]		
	AA Spectrophotometer		
24)	Does the spectrophotometer cover the wavelength range desired (suggested range of 190-880 nm)? [200-6.1; 200.7/200.9-6.1;SM3111 A]		
25)	Is the set-up (wavelength, slit width, burner or furnace head, and gasses) as recommended by the manufacturer? [200-9.1; 200.7/200.9-11.4; SM3113 B.4.b]		
26)	Is background correction being used? If not, are samples close to or above the regulatory limit reanalyzed with a nonabsorbing line to assess background contribution? [200-5.2.2; 200.7/200.9-11.4; SM3113B.2.a]		

		Y	N
27)	For 200.9, are corrected analyte signal, analyte conc. and background abs. capable of being displayed immediately for review and available as hard copy which is kept on file? [11.4.5]		
28)	For 200.9, are signals integrated only as peak area? [11.4.5]		
29)	Is autosampler being used for GFAA (furnace)? [200.9-6.1.5]		
30)	Is a mixture of hydrogen and argon gas supply used for analysis? Required for 200.9 [2.2]		
	ICP-AES [SM3120 & 200.7]		
31)	Is instrument computer controlled and capable of performing background correction? [SM-B.2.b; 200.7-6.1]		
32)	Can background be measured adjacent to analyte lines during analysis in a position free from spectral interference? [SM-B.2.b; 200.7-4.0]		
33)	Is high purity (99.99%) argon gas supply used for analysis? [SM-B.3.k; 200.7-6.1.3]		
	ANALYSIS -- General		
34)	Are reagent and standard solutions that have been prepared on site, dated and initialed by the analyst? NOTE: It is recommended that a log be maintained with the lot numbers of the chemicals used in each preparation. [Permit]		
35)	Are stock standards within expiration dates? [200-10.3.3; Permit]		
36)	Are Class A volumetric flasks used for preparing standards? [SM1070 B.2; Permit]		
37)	Are working calibration standards prepared by diluting stock metals solutions at the time of analysis for AA (200-8.2; SM3111 A.5); every 2 weeks for 200.9 [7.9]		
38)	Are calibration standards and blanks matrix matched to samples? (Identical conc of all acids used in digestion are in the standards and blanks.) [200-8.2; 200.7/200.9-7.9; SM3111 A.5/3113 B.4.c]		
39)	Are a minimum of 2 replicates of each sample, blank, check sample, etc. read? [200-8.2; SM3111 B.4.d; SM3120.B.4.c]		
40)	Has instrument detection limit been established? [SM 1030 E]		
	AA (FLAA and GFAA)		
41)	Has initial demonstration of performance been completed? [SM1020 B.1; 200.9-9.2.4]		
	a) Have quality control samples (QCS) - from different external source than standards - been analyzed with recovery of 90-110% of stated value?		
	b) Have method detection limits (MDL) been determined for each method and element? NOTE: MDL's must be determined annually using digested aliquots.		
42)	Is instrument stability determined prior to calibration by analyzing a standard solution with 20X conc of IDL a minimum of 5 times with resulting RSD of <5%? [200.9, 11.4.3]		
43)	Does the calibration curve consist of a blank and at least 3 standards and have a calibration coefficient of $\geq$ 0.995? [200-8.2 requires 4 Std.; SM1020 B.5]		
44)	Is instrument performance check (made from same source as standards) and calibration blank analyzed immediately after calibration (ICV/ICB), after every 10th sample (CCV/CCB), and at end of sample run (CCV/CCB)? Recoveries must be within 95-105% for ICV (200.9); 90-110% for ICV (other methods); 90-110% for CCV (all methods). [200-9.3.7; 200.9-9.3.4; SM3020]		
45)	Is a quality control sample (QCS) - from different external source than standards - analyzed to verify each calibration with a recovery of 90-110%? [200-10.3.4; 200.9-9.2.3; SM3020]		
46)	Is a laboratory reagent blank (LRB) analyzed with each batch of 20 or fewer samples of the same matrix? [200-4.1; 200.9-9.3.1; SM3020]		
47)	Is a laboratory fortified blank (LFB) analyzed with each batch of samples with a spike recovery of 85-115%? [200.9-9.3.2]		
48)	Are a minimum of 10% of samples being spiked prior to digestion? [200.9-9.4.2] For **GFAA** using SM or 200 series, is each sample site spiked until matrix consistency is demonstrated? [200-each method; SM3113]		

		Y	N
	a) Is recovery within 70-130% [200.9-9.4.3]; 85-115% [SM3111 A.7, 3113 B.4.d]?		
	b) If post digestion spike recovery is not within 85-115% is method of standard additions performed? [200.9-9.4.4.1; SM3020]		
49)	Are duplicate samples being analyzed at rate of 10% for 200 [10.3.50], 20% for SM [3020] with RPD ≤ 20%? (RPD's are valid only for values > 5X CRDL.)		
	ICP		
50)	Are all acids of trace metal grade or equivalent? [SM-B.3; 200.7-7.1]		
51)	Has initial demonstration of performance been completed? [SM1020 B.1; 200.7-9.2]		
	a) Has the upper limit of the linear dynamic range (LDR) been established for each method and element? [SM-B.1.b; 200.7-9.2.2]		
	b) Have quality control samples (QCS) - from different external source than standards- been analyzed with recovery 95-105% of stated value? [SM-B.4.e; 200.7-9.2.3]		
	c) Have method detection limits (MDL) been determined for each method and element? NOTE: MDL's must be determined annually. [SM1020 B.1; 200.7-9.2.4]		
52)	Have spectral interferences been documented? (This must be done initially and then annually thereafter.) [200.7-4.1.4, 7.13.5, and 10.4]		
53)	Is instrument calibrated according to manufacturer's instructions? [SM-B.4.b; 200.7-10.1]		
54)	Is a quality control sample (QCS) -- from different external source than standards -- analyzed daily with a recovery of 95-105% to verify calibration? [SM-B.4.e] QCS solution should be ≥1 mg/L, except silver, which must be 0.5 mg/L for solution stability. [200.7-9.2.3]		
55)	Is the interference check sample analyzed daily? [200.7-7.13]		
56)	Is a calibration blank used to flush the system after each solution (standards, samples, check solutions) is analyzed? [SM-B.4.d; 200.7-11.4.5]		
57)	Is an instrument performance check (made from same source as standards and in mid-range of curve) and calibration blank analyzed immediately after calibration (IPC/ICB), after every 10th sample (IPC/CCB), and at end of sample run (IPC/CCB)? Recoveries must be within 95-105% for first ICP; 90-110% for CCV. [200.7-9.3.4] 95-105% for all CCV's [SM-B.4.e]		
58)	Is a laboratory reagent blank (LRB) analyzed with each batch of 20 or fewer samples of the same matrix? [SM-B.4.d; 200.7-9.3.1]		
59)	Is a laboratory fortified blank (LFB) analyzed with each batch of samples with a spike recovery of 85-115%? [SM-B.4.f ; 200.7-9.3.2]		
60)	Are a minimum of 10% of samples being spiked prior to digestion? [200.7-9.4.2]		
	a) Is recovery within 70-130%? [200.7-9.4.3] 95-105% [SM-B.4.g]		
	b) If recovery is not within 70-130% is a post digestion spike analyzed with recovery of 85-115%, and a 1:4 dilution analyzed which agrees 90-110% of the original determination? [200.7-9.5]		
61)	Are duplicate samples being analyzed? (RPD's are valid only for values >5X the CRDL.) [SM3020; 200.7-9.4.1]		
62)	Are sample analyte concs > 90% of the LDR diluted and reanalyzed? [200.7-11.4.7]		
	DATA KEEPING		
63)	Are the permit levels met by the analytical technique chosen? [Permit]		
64)	Does raw data have analysts initials, analysis time and date recorded? [Permit]		
65)	Are all sample concentrations bracketed by standards? [Permit]		

		Y	N
66)	Are all raw data retained for at least 3 years? [Permit]		
67)	Is there a QA/QC plan available which includes a Corrective Actions section? [Permit]		

PROBLEMS:

Metals